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Title: Energetic Materials Formulations Advances

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Energetic Materials Formulations Advances

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The agglomeration process for producing plastic bonded explosives has been used for many years. However, until recently it has not been studied in detail to understand the mechanisms involved and to quantitatively determine the effects of the various control variables. A recent study of the slurry process to produce PBX-9501 used a small-scale (1-gallon) slurry reactor to quantify the effects on agglomeration by changing solvent removal rate, agitation speed, temperature, and sweep rate. The effects were determined quantitatively for the PBX-9501 process and are in good agreement with operational experience on larger reactor vessels. The study resulted in purely empirical functions, but future studies are planned to produce a theoretically-based agglomeration model.

Now an image analysis tool is being developed to measure particle size distributions (PSDs). The image analysis system will be used to measure PSDs during the slurry process as well as finished, dried molding powders. The PSDs of PBX 9501 library lots are currently being measured and plans are underway to see if empirical correlations exist between the PSD measurements and other measured properties of these library lots. Some preliminary results are presented.

Additional information on the current status of the TA-9 formulations buildings (under construction) and the planned formulation activities will be presented

2001 Energetic Materials Program Review: Advances in Formulations

DX-2 Engineering Team

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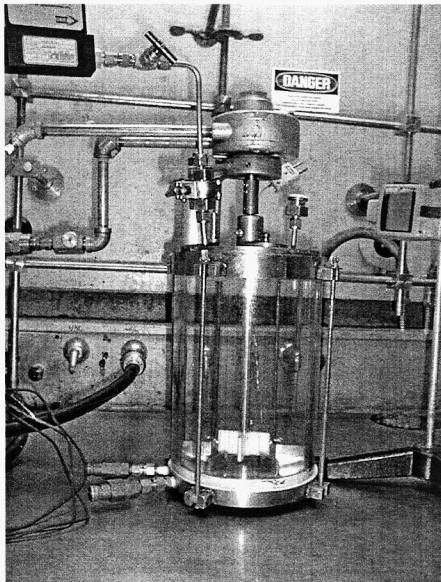
Gregg K. Sullivan (GRA)

Presentation Overview

- Current Formulation Capabilities
- Image Analysis Discussion
- PBX 9501 Library Lot Prill Size Study
- PBX 9501 Image Analysis Study
- Status of Process Buildings
- Future Work

Current PBX Formulation Capabilities

- Limited to lab scale (1 lb. Max)



- Recent Formulations Include PBX 9407, PBX 9501, & PBX 9502

Current PETN Recrystallization Capabilities

- Limited to lab scale (1 lb. Max)
- John Kramer Discussed

New HE Capabilities

- New explosives limited to lab scale (1 lb. Max)
- Mike Hiskey Discussed

Image Analysis Discussion: Measurements

- Length calculated
 - Use Area based (Waddel Diameter [D_w])
 - Others possible
- Circularity (ϕ) also calculated:
 - $4 * \pi * \text{Area} / \text{Perimeter}^2$
 - Examples:
 - Square, $\phi = 0.785$
 - Rectangle with $L/W = 10$, $\phi = 0.260$

Image Analysis Discussion: Pros and Cons

PROS

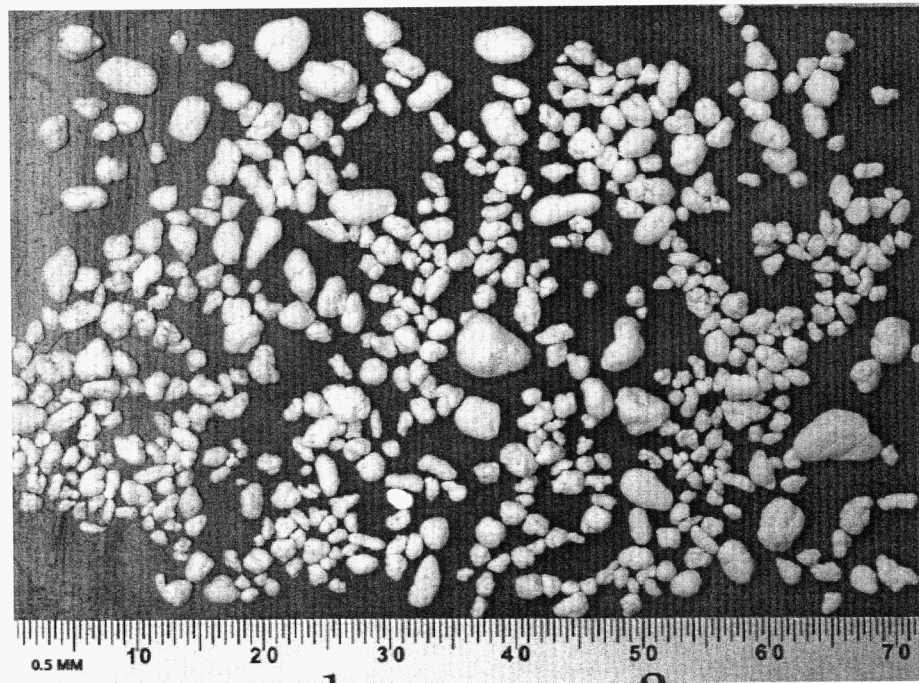
- Optics adjust for particle size
- Glass vessel adapted for imaging
- Off-the-shelf systems available

CONS

- Particle separation limited
- Image processing is slow
- Error and bias (discussed below)

Image Analysis Discussion: Dry Measurement Error and Bias

- Biased by gravity and particle shape
- Affected by sampling technique



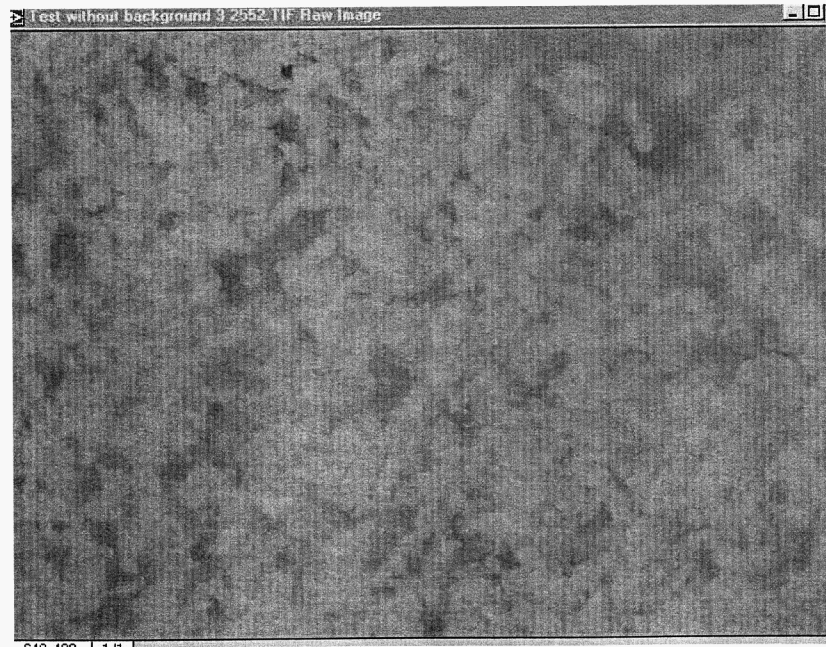
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Image Analysis Discussion: Slurry Measurement Error and Bias

- Fluid motion and pancaking bias measurement
- Particle concentration produces error



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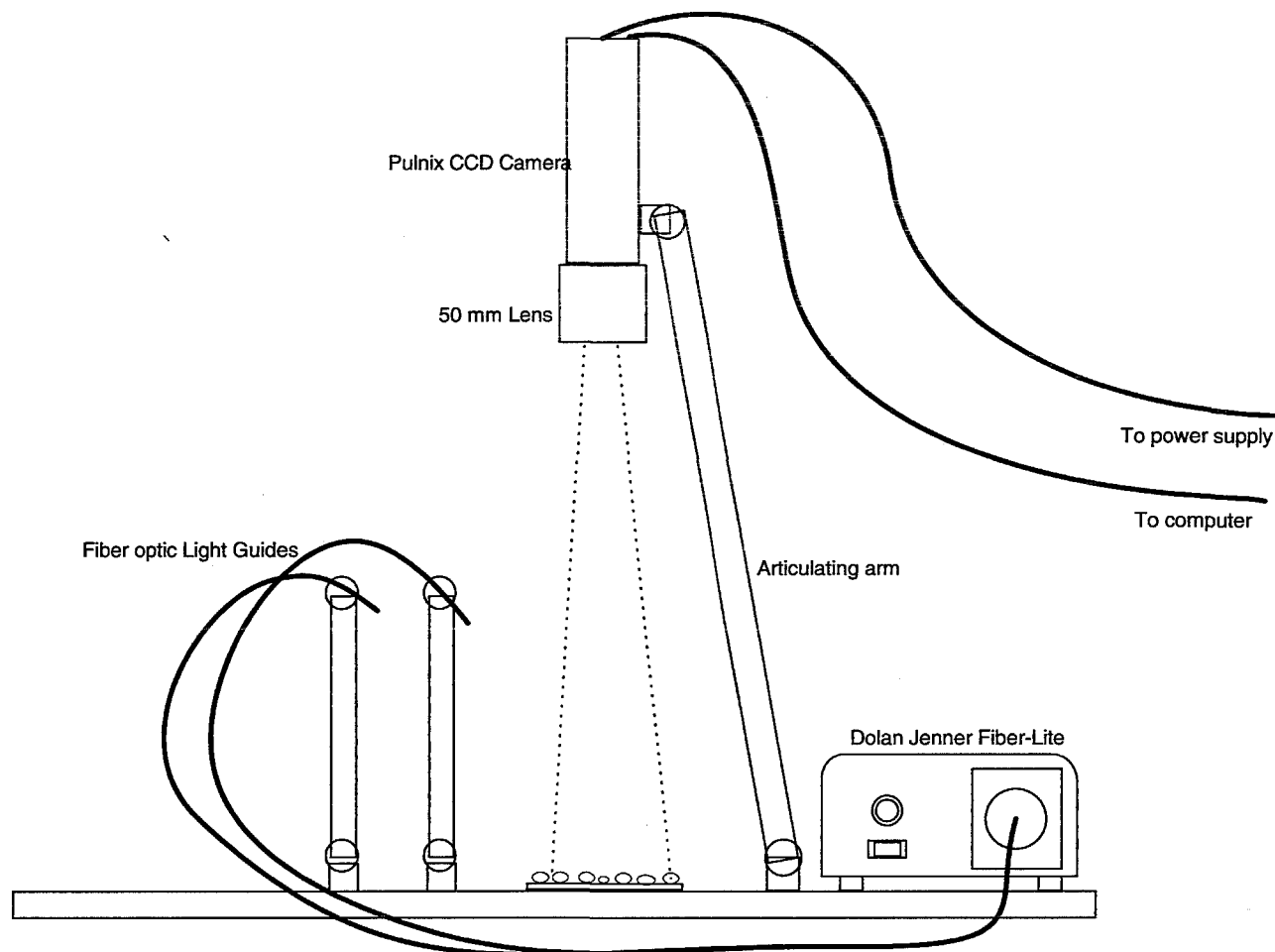
PBX 9501 Library Lot Prill Size Study: Introduction

- Observed pressing differences of unknown origin in latter 9501 lots.
- Application of Image Analysis Technology
- Provides comparative database for other PBX 9501 formulation efforts

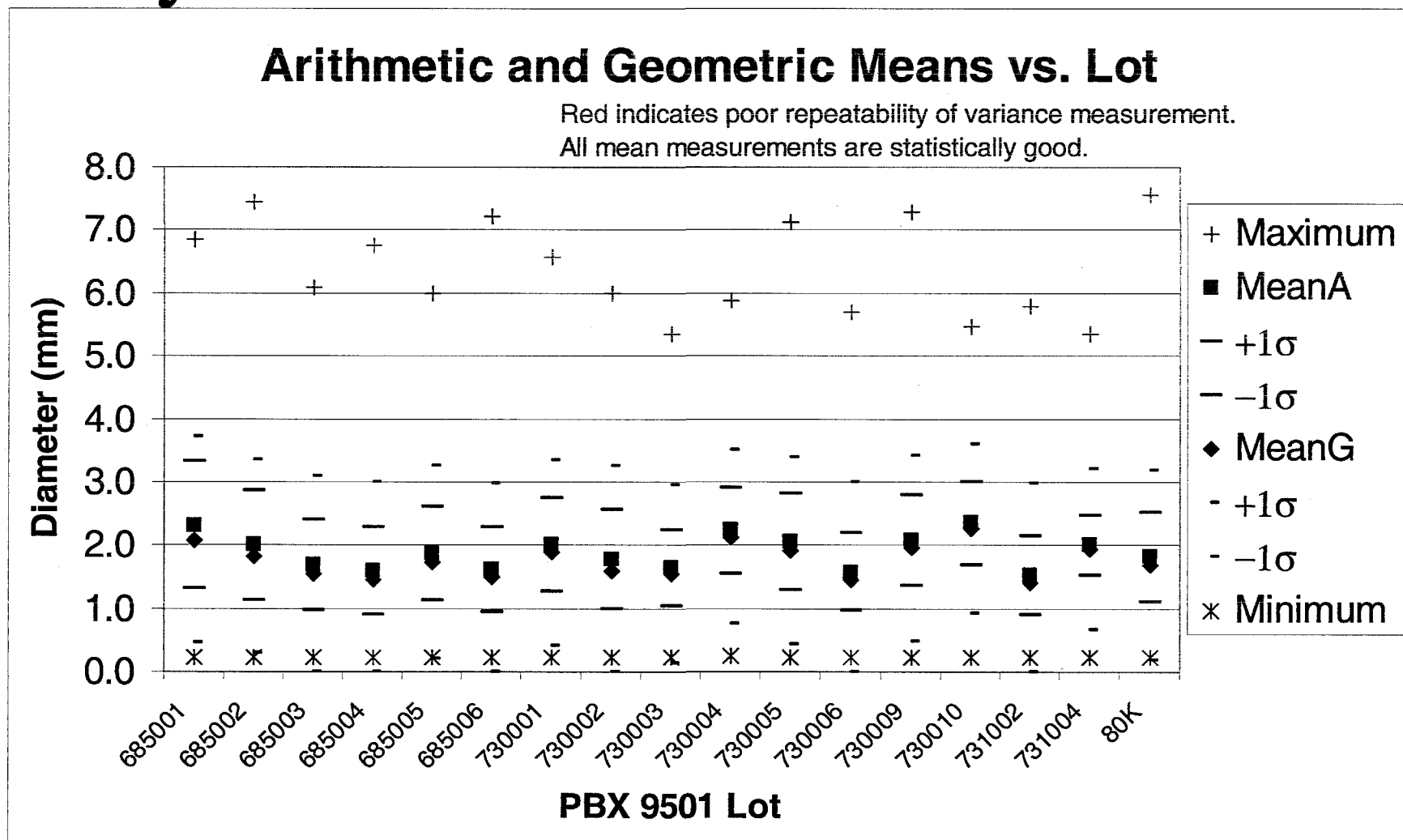
PBX 9501 Library Lot Prill Size Study: Approach

- Sample randomly and separate particles by hand
- Measure each lot three times and statistically test
- Examine other lot acceptance data and run correlation analyses

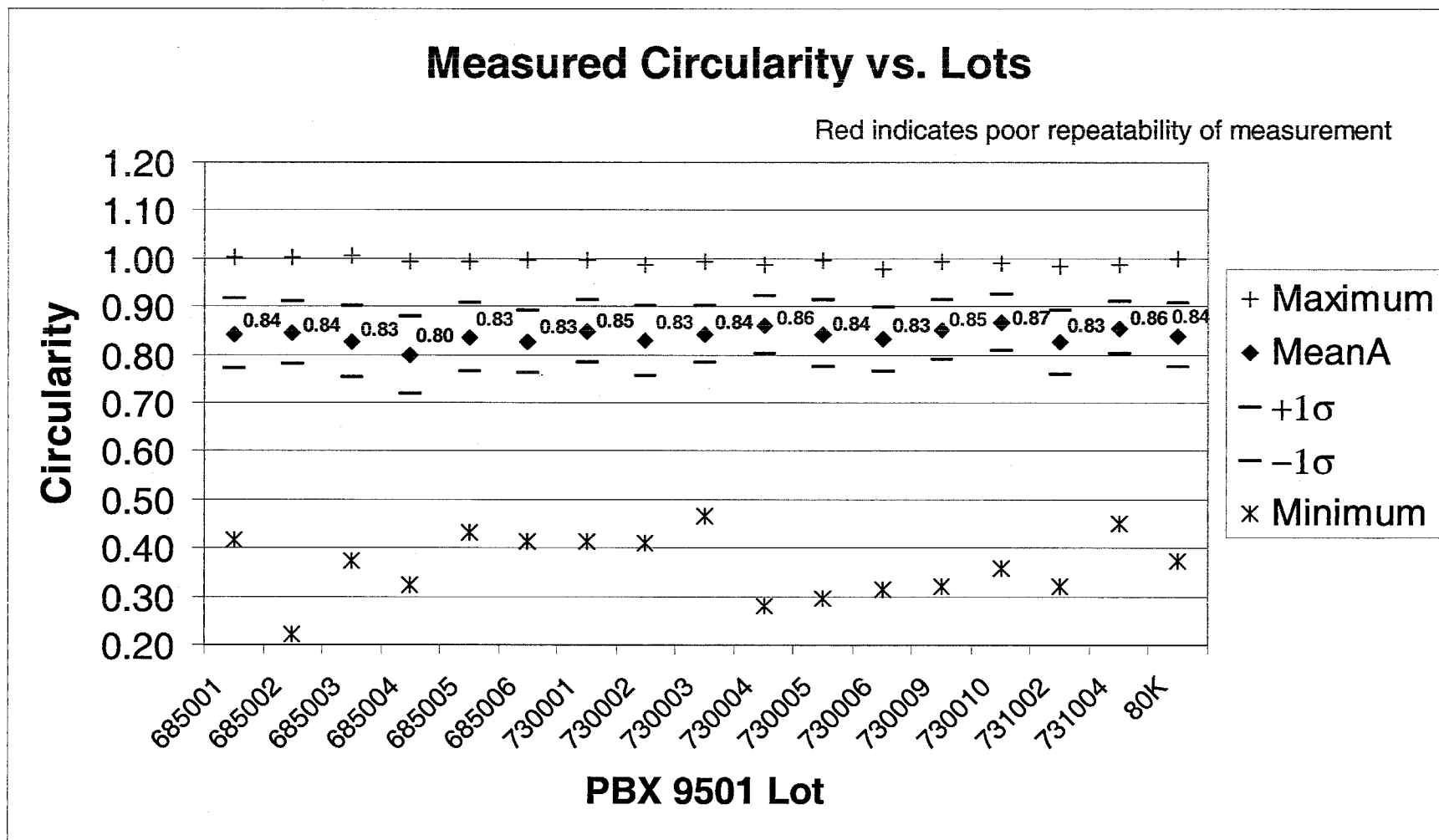
PBX 9501 Library Lot Prill Size Study: System



PBX 9501 Library Lot Prill Size Study: Results



PBX 9501 Library Lot Prill Size Study: Results

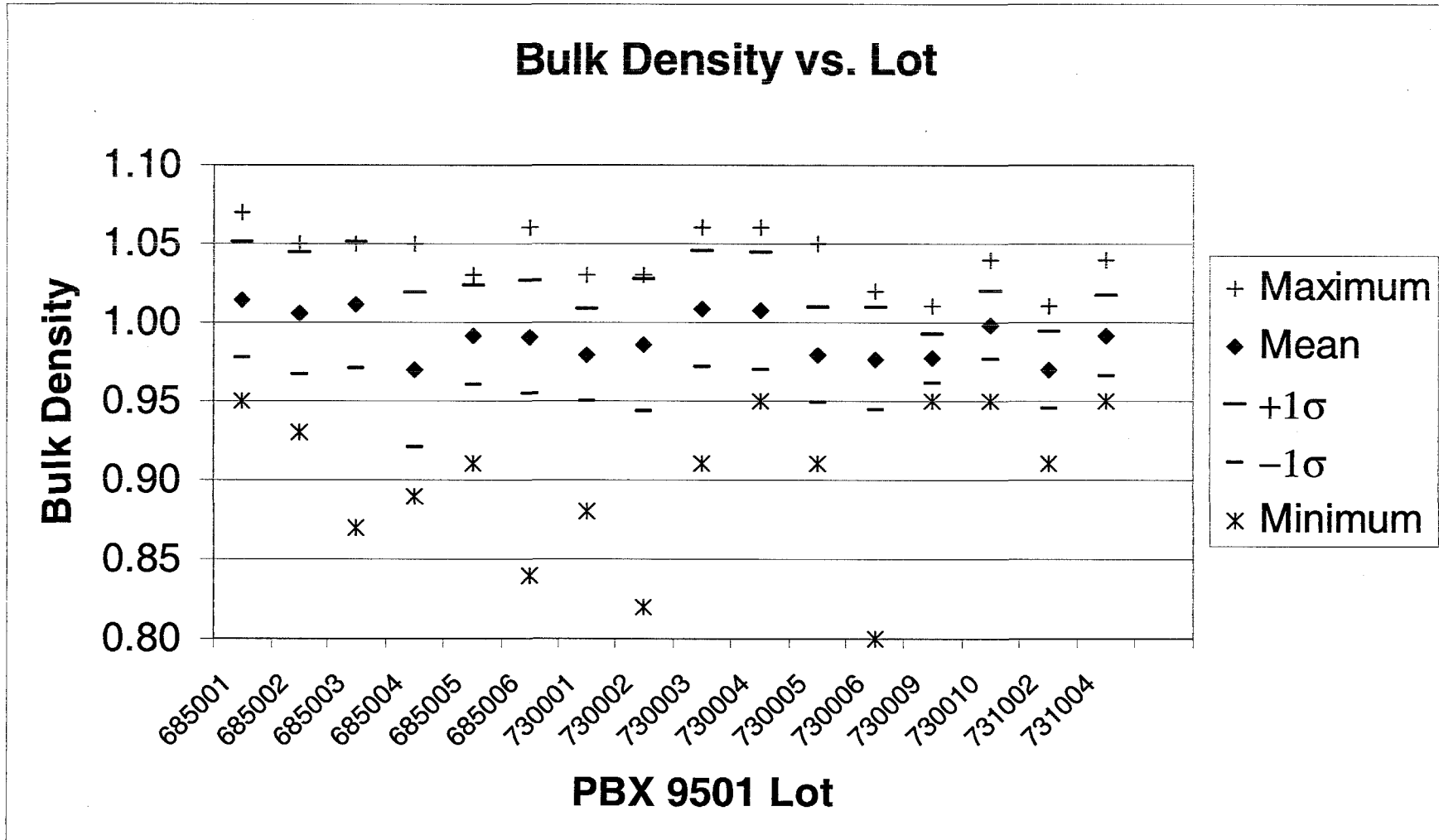


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PBX 9501 Library Lot Prill Size Study: Results

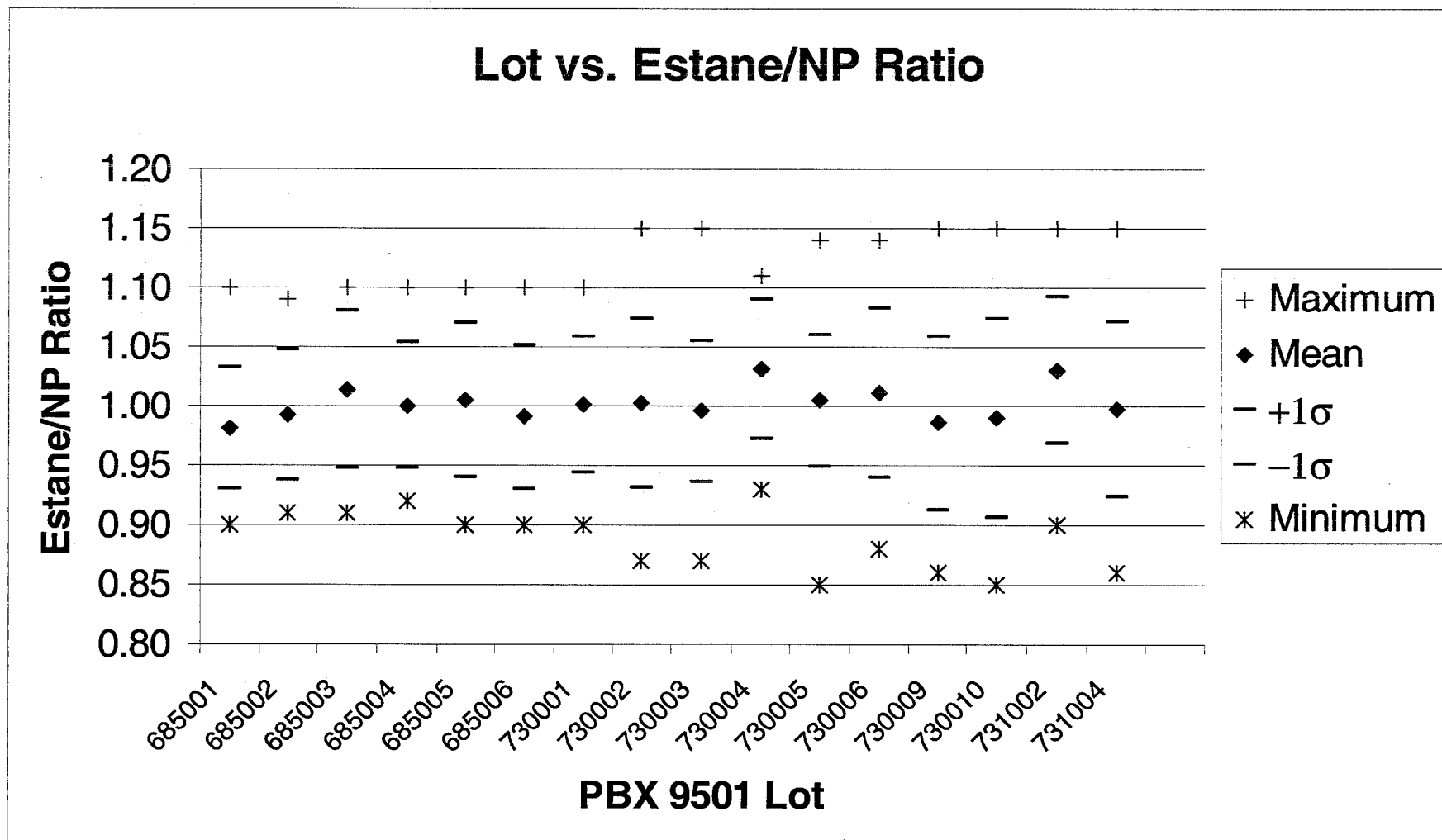


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PBX 9501 Library Lot Prill Size Study: Results

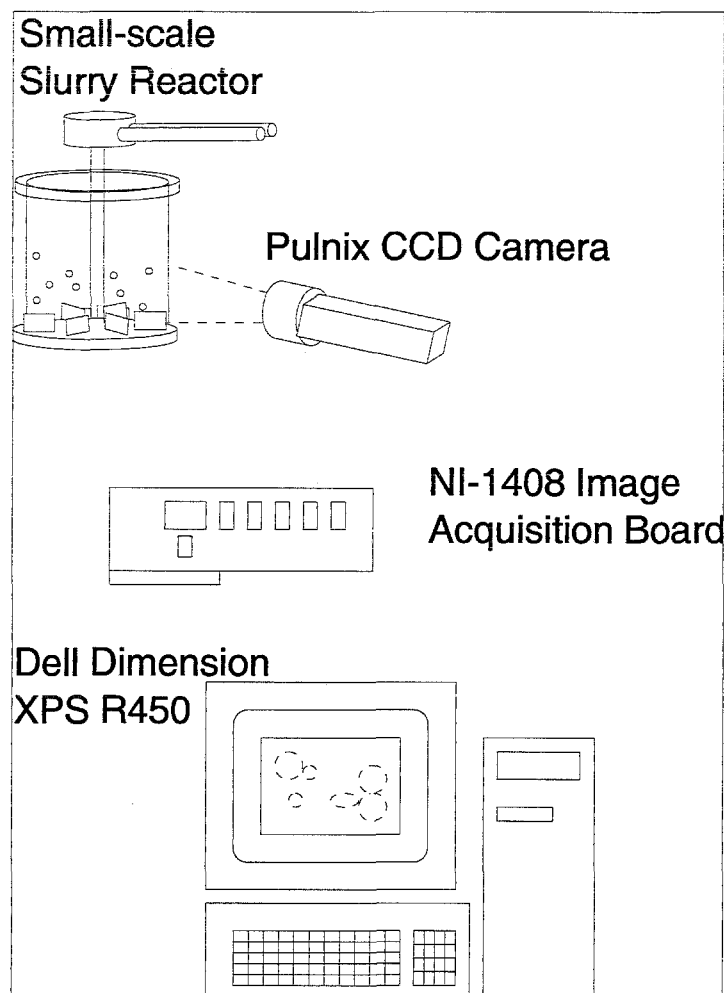


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Slurry Image System and Process Sequence



Snap Image

Equalize Image

Binarize Image

Separate Particles

Measure Particles

Calculate Distribution

Slurry Measurements and Variables

■ Independent Variables

- Temperatures [$^{\circ}\text{C}$] (vessel, hot glycol & cold glycol)
- Sweep Rate (SLPM)
- Agitation (RPM)
- Pressure (kPa)

■ Dependent Variables

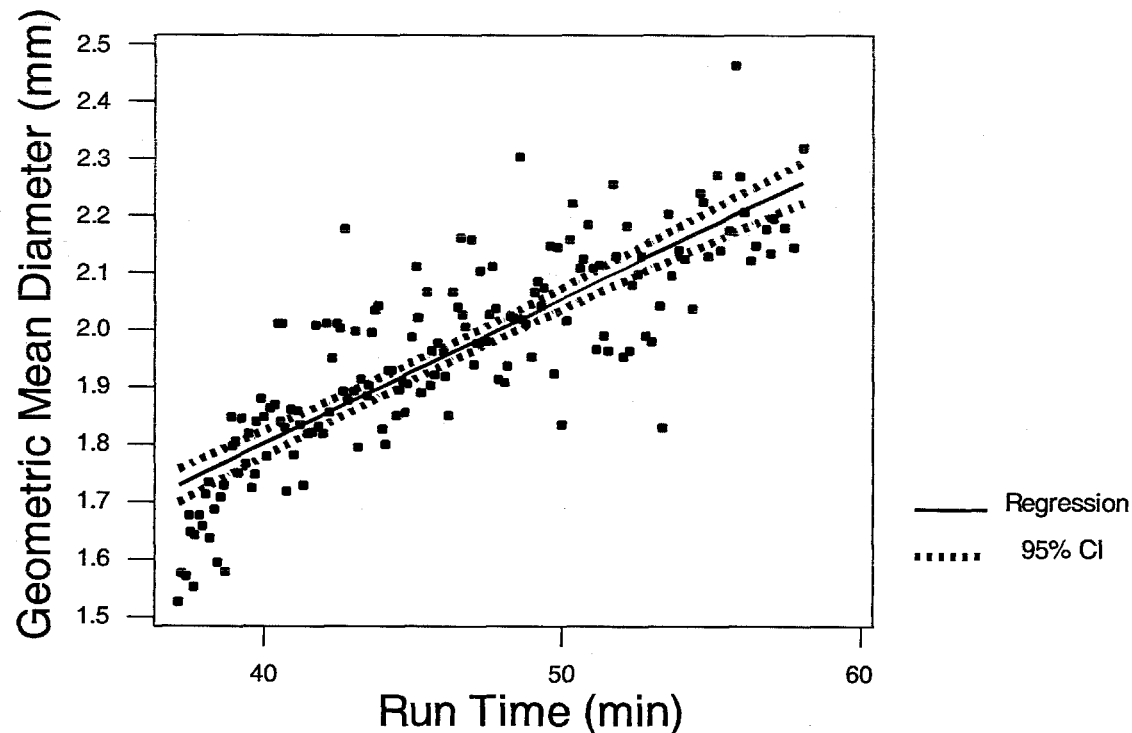
- PSD
- Solvent Mass (g) Removal Rate (g/time)
- Continuous Phase Solvent Level (mass fraction)

Slurry Measurement Preliminary Results: No Particle Deflector

Regression Plot

$$Y = 0.787534 + 2.53E-02X$$

R-Sq = 68.4 %

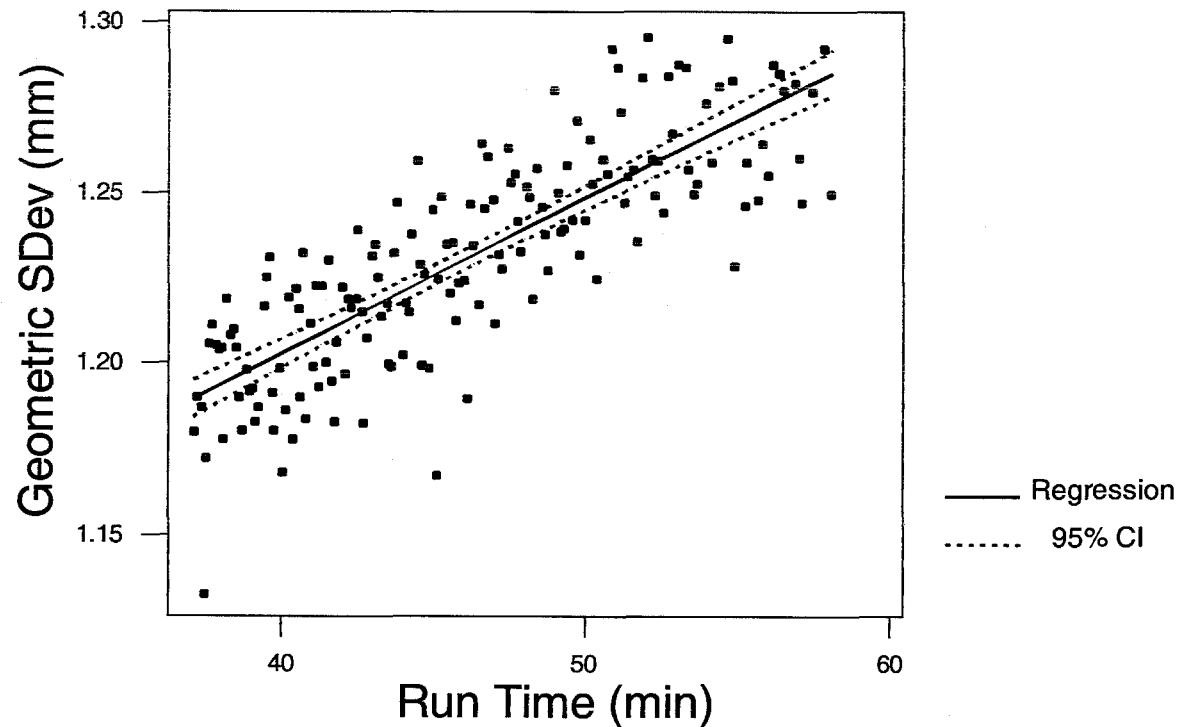


Slurry Measurement Preliminary Results: No Particle Deflector

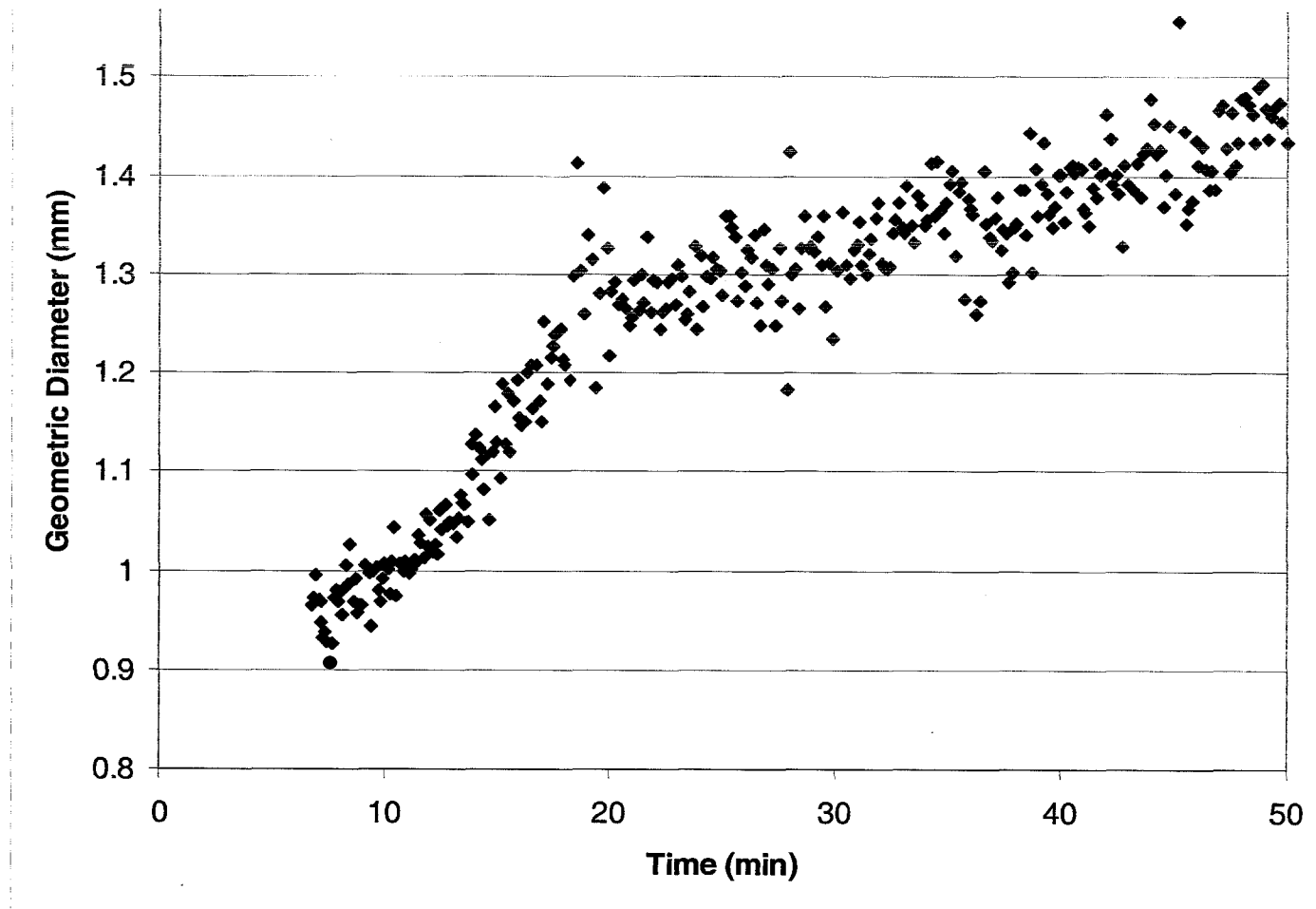
Regression Plot

$$Y = 1.02144 + 4.54E-03X$$

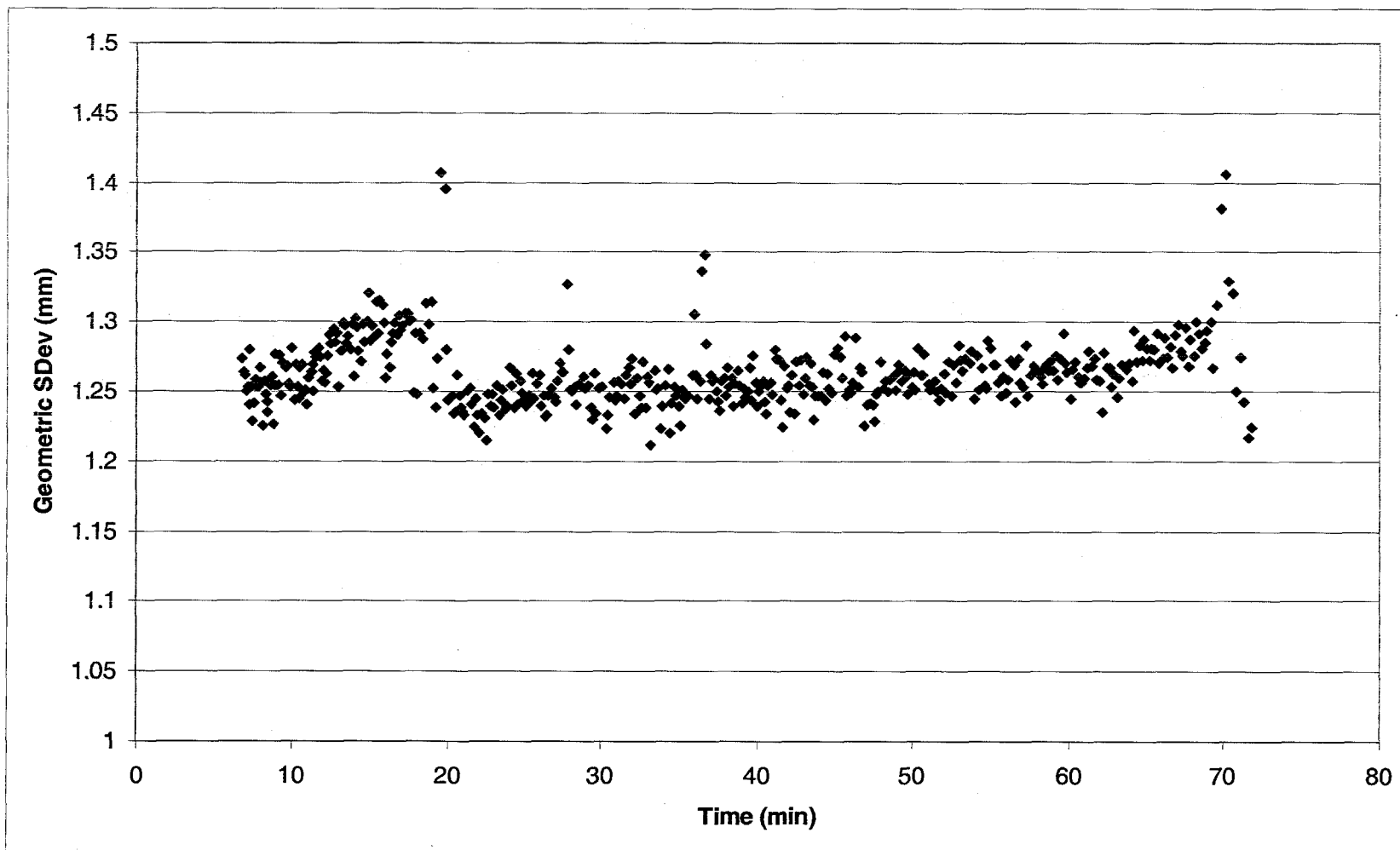
R-Sq = 66.5 %



Slurry Measurement Preliminary Results: With Particle Deflector



Slurry Measurement Preliminary Results: With Particle Deflector



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Slurry Experimental Test Matrix

| Factor | Factor Levels |
|------------------|----------------|
| Temperature (°C) | 35, 45, 55 |
| Flow Rate (SLPM) | 5, 12.5, 20 |
| Agitation (RPM) | 600, 900, 1200 |

- Each experimental point is repeated 3 times
- Experiments are randomly ordered

First Slurry Experimental Point Results

- TBD like the earlier preliminary plots

First Slurry Experimental Point Results

- TBD Like the earlier preliminary plots

Some Additional Test Points

- Two additional tests have been proposed:
 - Shut in system and look for growth rate with constant solvent level
 - Start with dry molding powder, add solvent in increments, and measure the breakup rate.
- Represents a total of six additional points

Remaining Work

- Complete PBX 9501 library lot correlation analyses
- Complete slurry experiments and modeling
- Scale up

Formulation Building Status

- Building 38
- Building 45
- Building 46

Future Formulations

- Fibers in PBX 9501
- Latex Binders
- PETN recrystallization efforts
- Hiskey's new HE
- MIC Formulations